

INFLUENCE OF GEL FORMATION TEMPERATURE AND TIME ON COMPLETE CONVERSION OF FLY ASH IN TO A FRAMEWORK ALUMINOSILICATE UTILIZING ALKALINE HYDROTHERMAL SYNTHETIC METHODOLOGY

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ABSTRACT

The use of coal, which has the largest amount of deposit as an energy source and its use, is significantly increasing every year in the electric power plants. Coal fly ash discharged from coal electric power plants occupies a great part of the total amount of ash. In India fly ash is being generated at the rate of about 60 million tons per annum (MTPA) from about 75 thermal power plants and is expected to increase to 100 MTPA by the turn of the century. About half of the discharged fly ash is used as a raw material for cement and so on and rest of it is disposed to landfill site. There are serious environmental health hazards associated with fly ash. In addition, land requirement envisage for disposal of fly ash is about 50,000 acre with an annual expenditure of about Rs500 million for transportation. To overcome these difficulties a number of technologies have been developed for gainful utilization of fly ash which ranges from low to high value added applications. India records a very low percentage of 15-20% as compared to a corresponding figure for developed countries. In this aspect, the possibility of synthesizing high value products such as zeolites from fly ash was explored.

KEYWORDS: Fly Ash, Zeolite, Hydrothermal Synthesis, Waste Utilization, Value Added Products

INTRODUCTION

Coal based thermal power plants plays a pivotal role in supplying nation's ever-increasing demand for electricity. World net electricity generation is expected to increase by 77% from 2006-2030 and coal continues to fuel the largest share and remains the least expensive of all the fossil fuels for electricity generation worldwide with total hard coal consumption of 5522 MT (million tons) [01]. About 65% of the total electricity generation is fuelled by coal and is projected to increase 5 folds in 25 years [02]. Indian coal is generally non-coking coal and is of poor quality in terms of high ash content (ranging from 30-35%), high moisture content (ranging from 4-20%), low calorific value (between 2500-5000 kcal/kg) and high amounts of toxic trace elements which leads to more use of coal for the generation of same amount of electricity and a large quantity of waste (fly ash, FA), with the estimated global production of 200 million tones per year [02-04]. In India, about 70% power plants are coal based [03, 05] which generate more than 112 million tones of fly ash annually and it is projected to increase up to 160 million tones per year [03, 06]. **Therefore, there is an urgent need to encourage interdisciplinary research on the development of materials using fly ash through novel synthetic methodologies.** Less amount of beneficial nutrients, low cation exchange capacity, high alkalinity, presence of hazardous leachable trace elements and high transport cost for relatively low value product are some factors responsible for the limited utility of fly ash [07]. Many technologies have been developed for gainful utilization of fly ash ranging from low to high value added applications [08]. Some of the examples include the use of fly ash as a bottom layer in road construction, in concrete and ceramic production, in building industries and also as backfills [09]. **About 5-6% of total fly ash is used up in the above**

mentioned applications, and rest of it is disposed off in landfill sites and ash pools which is not only expensive but also causes serious environmental and health problems (air, water and soil pollution) [05, 10]. Annual expenditure for the disposal of fly ash to ash dams (~ 65,000 acres of land) is more than Rs. 500 million. Several attempts have been made for proper utilization of fly ash, either to reduce the cost of disposal or to minimize its impact on environment [07]. Increasing concerns about the environmental consequences of such disposal have forced us to put our efforts in the direction of cost effective process development for the conversion of fly ash to value added product/s and its utilization in various commercial processes.

It has therefore become necessary to look at the method that can be used to produce value added products from fly ash. For the last few years there has been much interest in zeolites. Synthesis of zeolites using fly ash is a well-known process, which may resolve the disposal problem, thereby minimizing its effect on the environment. The fly ash contains more than 80% silica and alumina and can serve as the raw material for synthesis of zeolites. High cation exchange capacity, high surface and variable pore size are some of the special features which make zeolites versatile materials for targeting wide range of applications e.g., materials for soil remediation and enhancement of plant growth, catalysts for chemical and petrochemical industries, adsorbents for a wide array of cationic and anionic pollutants, as ion-exchanger for nuclear wastes treatment etc.

Therefore, cost effective production of zeolites using coal fly ash constitutes one important issue of waste management. Several research reports have been published on synthesis of zeolites [11] (P, A, X, Y, analcime, chabzite etc) using various synthetic procedures such as hydrothermal method, hydrothermal with closed system method, fusion method, refluxing method, microwave assisted methods [07, 08, 12-25]. These attempts are beset with drawbacks of low crystallinity, lower yield, residual fly ash in the product, more than one products etc.

Therefore it is of utmost importance to develop cost effective process development for the total conversion of fly ash to a single and value added product and its utilization in various commercial processes.

EXPERIMENTAL

Materials and Methods

In the present investigation, fly ash sample was procured from National thermal Power Plant, India and sodium hydroxide (AR, 98%) Pure was procured from Qualigens, India. The aluminosilicate from fly ash was synthesized by the modification of the reported procedure [15].

Synthesis

In order to convert fly ash into a value added product, the complete dissolution of the fly ash, in the form of a gel, has been done by heating the alkali fly ash mixture. After the formation of the gel, the solution was kept for ageing step (close to the room temperature) followed by the curing /crystallization step as per the reported literature. 5g of fly ash sample was taken; fly ash sample was sieved to eliminate larger particles. The fly ash was added to sodium hydroxide solution in a conical flask to make the slurry and was stirred at various temperatures followed by ageing and curing. The resultant mixture was then cooled to room temperature and then washed several times with double distilled water to eliminate extra alkali followed by drying in oven at 60-70°C for 6-7 hours.

Keeping in mind the need for the synthesis of a value added product and cost effectiveness of the process, effect of parameters such as gel formation temperature and time was studied on the above mentioned method by keeping the entire parameters (alkali concentration, ageing temperature and time and curing temperature and time) constant. In order to verify the reproducibility of the procedure, four batches of samples were synthesized using above mentioned conditions. Each sample was analyzed by all relevant techniques to identify the structure of the synthesized product.

Characterization

Various analytical techniques have been employed for this purpose. To identify crystalline materials in the samples, X-ray diffractograms were recorded on Philips PW3710 X-ray Diffractometer using Cu K α (alpha) radiations with tube voltage 45 kV and 40 mA with a sampling step of 0.02° and a scan time of 4sec in 2 θ values ranging from 10-70°. Infrared spectroscopic (FT-IR) studies were carried out to identify their structural features. FTIR spectra were taken in KBr on a Perkin Elmer FTIR spectrophotometer. For each sample, spectrum has been recorded for 64 scans with 4cm⁻¹ resolution between 4000–400cm⁻¹ region. To avoid the interference from the CO₂ and water, IR chamber was flushed with dry nitrogen. Morphology of fly ash and synthesized product were investigated by using scanning electron microscope (SEM), the SEM analysis has been performed on ZEISS EVO Scanning Electron Microscope Model EVO 50. Thermal stability was investigated by thermo gravimetric (TGA) methods. TGA was recorded on Shimadzu DTG 60 at a heating rate of 30°C/min upto 700°C and a flow rate of 100mL/min.

PRODUCT PERFORMANCE AS A FUNCTION OF THE GEL FORMATION TEMPERATURE

The chemical and physical nature of any reaction product is highly dependent on the reaction parameters. Therefore, in order to develop a novel method for the conversion of fly ash into a value added product/s, product performance has been monitored as a function of variation in gel formation temperature and time.

Variation in Initial Mixing Temperature

Keeping 3M NaOH as the initial concentration of alkali, initial mixing temperature was varied from 30 to 80 °C (“Table 1”).

RESULTS AND DISCUSSIONS

X-Ray Diffraction Studies

The x-ray powder diffraction patterns (“Figure 1” a, b, c and d) of fly ash (S-01) and the synthesized products (S-02, S-03 and S-04) indicates the conversion of fly ash to crystalline aluminosilicates. A relatively broad band centered at 2 θ = 26.2° is the characteristic feature of class F fly ash (glassy phase) having relatively low calcium content (“Figure 1” a) [26, 27]. The presence of sharp peaks at 2 θ = 16.34 (5.41), 30.81 (2.89), 33.11 (2.70), 35.12 (2.55), 39.13 (2.29), 40.73 (2.21), 60.50 (1.52) and at 2 θ = 20.77 (4.27), 36.79 (2.44), 49.99 (1.82), indicates the presence of predominantly mullite (3Al₂O₃·2SiO₂) and quartz (SiO₂) respectively, as the crystalline material (JCPDS no. 15-0776 and 05-0490). The halo pattern in the background between 2 θ = 10.2° and 2 θ = 40.2° indicates the presence of amorphous material [28].

Appearance of increasing amount of crystalline phase/material with the increasing initial mixing temperature and the disappearance of diffraction peaks of fly ash (“Figure 1” b-d) is indicative of gradual increase in the extent of conversion of fly ash in to crystalline aluminosilicate. The diffraction intensity at 2 θ = 13.605, 2 θ = 22.56 (3.93), 30.37(2.94) and at 2 θ = 26.75 (3.32), was found to match with hydroxysodalite structure, faujasite and quartz respectively

(12-0228 and 05-049) ("Figure 1" b). Thus, the sample is the mixture of zeolites and fly ash. The disappearance of diffraction peaks of faujasite is indicative of gradual increase in the extent of conversion of fly ash in to crystalline aluminosilicate, in case of sample S-03 ("Figure 1"). The presence of quartz peak at $2\theta = 26.53$ (3.35) shows the presence of fly ash in the sample. The increase in the intensity counts and the disappearance of diffraction peaks of fly ash and faujasite ("Figure 1"), suggests the formation of the single crystalline cubic structure.

Fourier Transformed Infrared Spectroscopic Studies

The FTIR spectra of ("Figure 2" a, b, c and d) of fly ash (S-01) and the synthesized products (S-02, S-03 and S-04) indicates the conversion of fly ash to crystalline aluminosilicates. Absence of representative peaks of fly ash ("Figure 2" a and "Table 2") [29], in the FT-IR spectra of the synthesized product, S-02, S-03 and S-04 ("Figure 2" b-d) suggests total conversion of fly ash. The strongest band appears at 717, 666 and 561 cm^{-1} in case of the sample S-02 ("Figure 2" b), are in good agreement with the literature reported bands of the faujasite [30]. The increase intensity of the bands of hydroxysodalite and the disappearance of peaks of faujasite in sample S-03 ("Figure 2" c), is indicative of gradual increase in the extent of conversion of fly ash in to crystalline aluminosilicate. Three well-defined medium intensity bands at 718, 696, 661 cm^{-1} and the bands at 463 and 434 cm^{-1} has been observed for the S-04 sample ("Figure 2" d) and are in good agreement with the bands reported in the literature for hydroxysodalite structure [31,32]. The absence of the band because of the double 6 ring, at 555 cm^{-1} , also supports the hydroxysodalite formation [30].

Scanning Electron Microscopic Studies

Scanning electron micrographs of the fly ash (S-01) and the corresponding synthesized products (S-02, S-03 and S-04) indicates the conversion of fly ash to crystalline aluminosilicates ("Figure 3"). The SEM picture of the fly ash ("Figure 3" a) indicates smooth spherical surface [27] with particle size ranging from 2-5 μm . Appearance of deformed spherical particles and some cubic structure is indicating the presence of residual fly ash and faujasite in the case of sample S-02 ("Figure 3" b). The appearance of sharp edges on the surface is clearly seen and the absence of cubic shapes suggesting the conversion of fly ash in to the single crystalline material in sample S-03 ("Figure 3" c). Decrease in particle size and a well defined cubic morphology in case of S-04 sample ("Figure 3" d) indicates the complete conversion of fly ash in to a crystalline product.

Thermogravimetric studies

Thermograms of fly ash and the synthesized product indicates the conversion of fly ash to crystalline aluminosilicates ("Figure 4" a, b, c and d). In case of fly ash ("Figure 4" a) first weight loss of 0.2% is observed in the temperature range of 35-200°C (corresponding to the loss of physically adsorbed water) followed by a weight loss of 1.8% in the temperature range of 200-550°C (attributed to the decomposition of hydrated salts such as $\text{Ca}(\text{OH})_2 \cdot x\text{H}_2\text{O}$, $\text{CaSO}_3 \cdot x\text{H}_2\text{O}$, etc. present in fly ash). In the temperature range of 550-700°C 0.8% weight loss (attributed to the loss due to the oxidation of unburnt carbon and decomposition of metal carbonates in fly ash) has been observed [33, 34].

Two step weight losses in each case were observed for the samples S-02, S-03 and S-04. The first step, 30°C to 100°C, amounts to a loss of 3.2%, 1.8% and 3.1% corresponds to the loss of physically adsorbed water. The second step, 100°C to 250°C, amounts to a loss of 6.6%, 2.4% and 4.9% for S-02, S-03 and S-04 respectively ("Figure 4" b-d) corresponds to the loss of metal bound water and water located in zeolitic cavity [35]. The total percentage of weight loss in case of fly ash has been relatively very small (2.8%) due to non-framework structure. The presence of cavities and voids

in the framework is responsible for the retention of relatively large amount of water in synthesized products (S-02, S-03 and S-04).

Variation in Initial Mixing Time

Keeping 3M NaOH as the concentration of alkali and 80 °C as the gel formation temperature, gel formation time was varied from 24hrs to 48 hrs ("Table 3").

RESULTS AND DISCUSSIONS

X-Ray Diffraction Studies

The x-ray powder diffraction patterns ("Figure 5" a, b and c) of fly ash (S-01) and the synthesized products (S-05 and S-06) indicates the conversion of fly ash to crystalline aluminosilicates.

The diffraction intensity at $2\theta = 30.78$ (2.90) and at $2\theta = 13.4$ (6.60) and 23.62 (3.76), was found to match with faujasite and hydroxysodalite structure ("Figure 5"b). The disappearance of diffraction peaks of faujasite and the increase in the intensity counts of diffraction peaks of hydroxysodalite in the case of sample S-03 ("Figure 5" c), is indicative of the formation of the single crystalline cubic structure. The diffraction intensities in the XRD patterns were found to be in good agreement with hydroxysodalite structure [31, 32].

Fourier Transformed Infrared Spectroscopic Studies

The FTIR spectra of ("Figure 6" a, b and c) of fly ash (S-01) and the synthesized products (S-05, and S-06) indicates the conversion of fly ash to crystalline aluminosilicates. The strongest band appears at 987 and 989 cm^{-1} in case of the synthesized product S-05 and S-06 ("Figure 6" b), are in good agreement with the literature reported bands of the faujasite. Three medium intensity bands at 718 , 696 , 661 cm^{-1} and the bands at 463 and 434 cm^{-1} has been observed for the S-06 sample ("Figure 6" c) and are in good agreement with the literature reported bands reported for hydroxysodalite structure [09]. Appearance of increase intensity of the bands of hydroxysodalite ("Figure 6" b and c) is indicative of gradual increase in the extent of conversion of fly ash in to crystalline aluminosilicate. The absence of the band because of the double 6 ring, at 555 cm^{-1} , also supports the hydroxysodalite formation [30].

Scanning Electron Microscopic Studies

Scanning electron micrographs of the fly ash and the corresponding synthesized products (S-01, S-05 and S-06), ("Figure 7" a-c) indicates the conversion of fly ash to crystalline aluminosilicates. Appearance of the sharp edges on the spherical surface is clearly seen in the case of sample S-05 ("Figure 7" b), suggesting higher conversion of fly ash in to the crystalline material. In case of S-06 sample ("Figure 7" c), a well-defined cubic morphology indicates the complete conversion of fly ash in to a crystalline product. The XRD, FTIR data is also supportive of this observation.

Thermogravimetric Studies

The thermo-grams ("Figure 8" a, b and c) of fly ash and the synthesized products (S-01, S-05 and S-06) indicates the conversion of fly ash to crystalline aluminosilicates.

Two step weight losses in each case were observed for the samples S-05 and S-06. The first step, 30°C to 100°C , amounts to a loss of 2.8% and 3.1% and the second step, 100°C to 250°C , amounting to a loss of ~5.2% and ~4.9% for S-05 and S-06 respectively ("Figure 8" b and c).

CONCLUSIONS

From the characterization studies of fly ash and the alkali modified fly ash, it is concluded that Pure form, single phase and highly crystalline zeolite hydroxysodalite samples were synthesized and it has been found that the best product is obtained with gel formation temperature of 80°C and stirring time of 48 hours.. **The structure was found to have cubic type structure.** Present process formulated here could open a new and alternative approach for the massive recycling of coal fly ash. This process has the advantages of cost effectiveness, technically convenient, economical and non-tedious process, high value utilization. So, the outcome of the proposed research work would lead to a cleaner and healthier environment. Therefore, further work was carried out using 3M NaOH as the initial concentration of alkali with 80°C as the initial stirring temperature and stirring time of 48 hours.

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APPENDICES

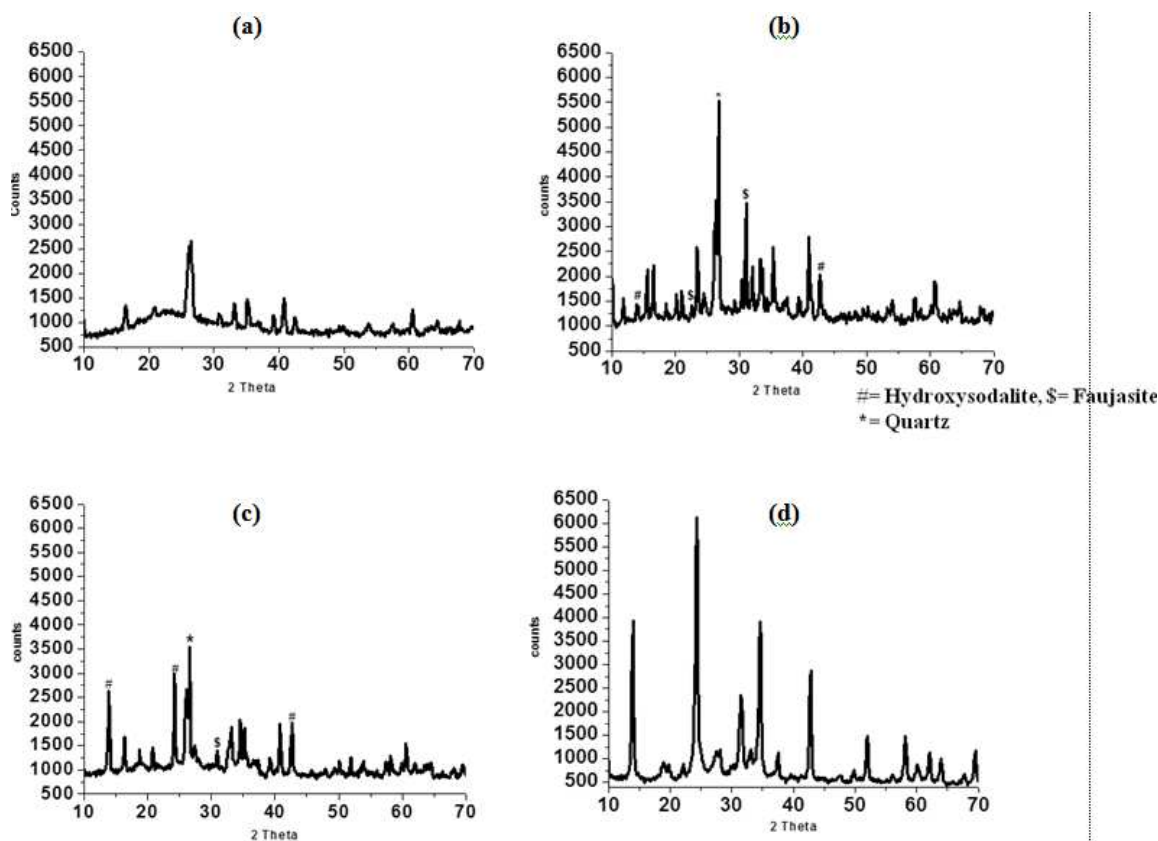


Figure 1: XRD Patterns of Fly Ash and the Product Obtained at Varying Temperature of Gel Formation

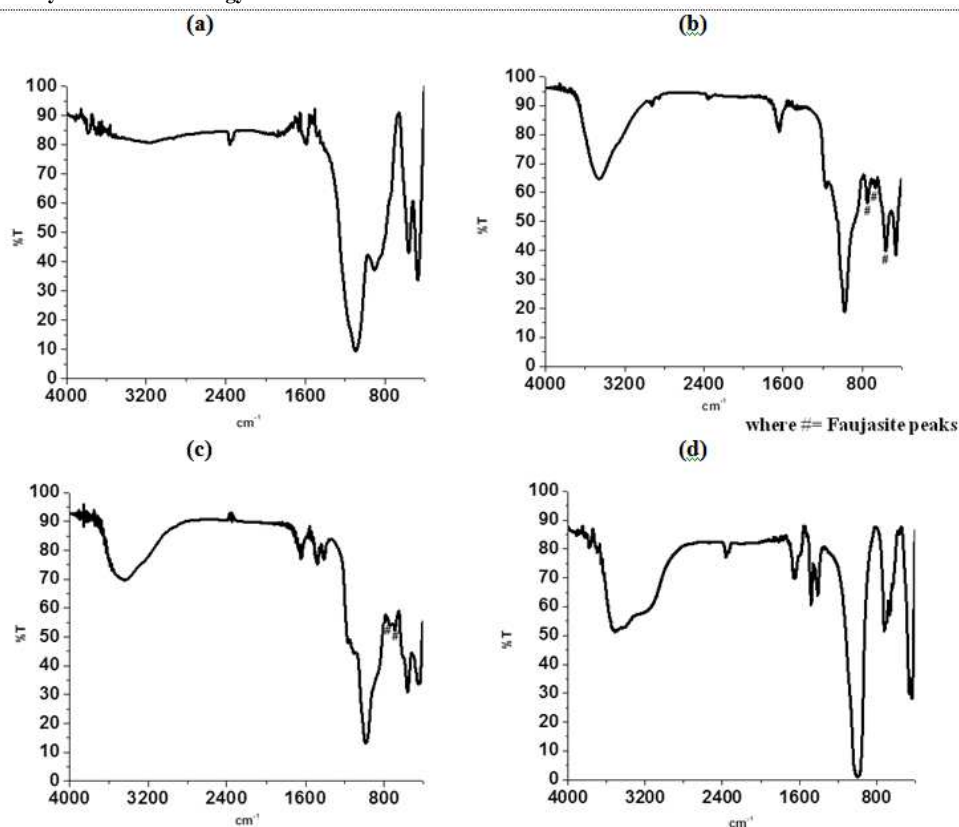


Figure 2: FT-IR Patterns of Fly Ash and the Product Obtained at Varying Temperature of Gel Formation

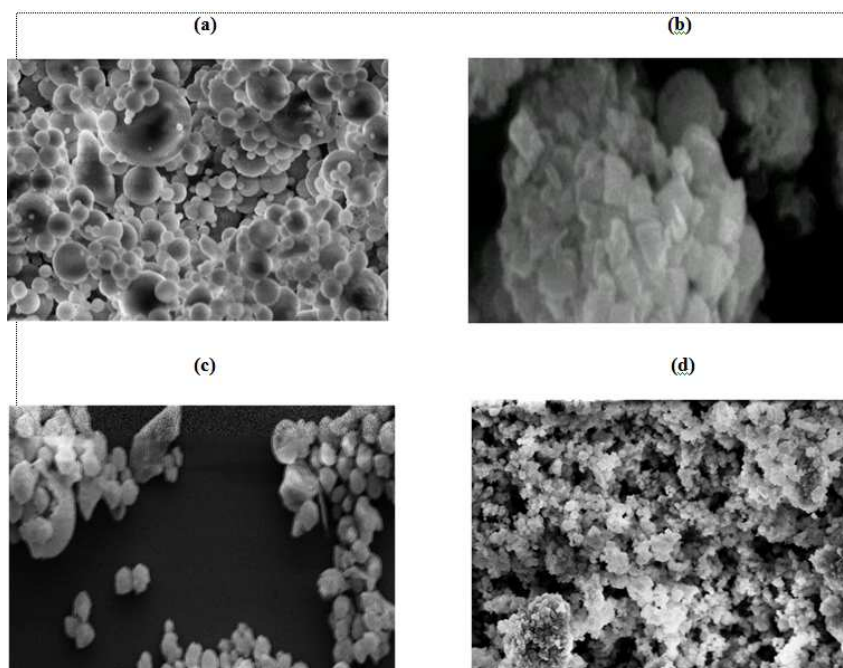


Figure 3: SEM Patterns of Fly Ash and the Product Obtained at Varying Temperature of Gel Formation

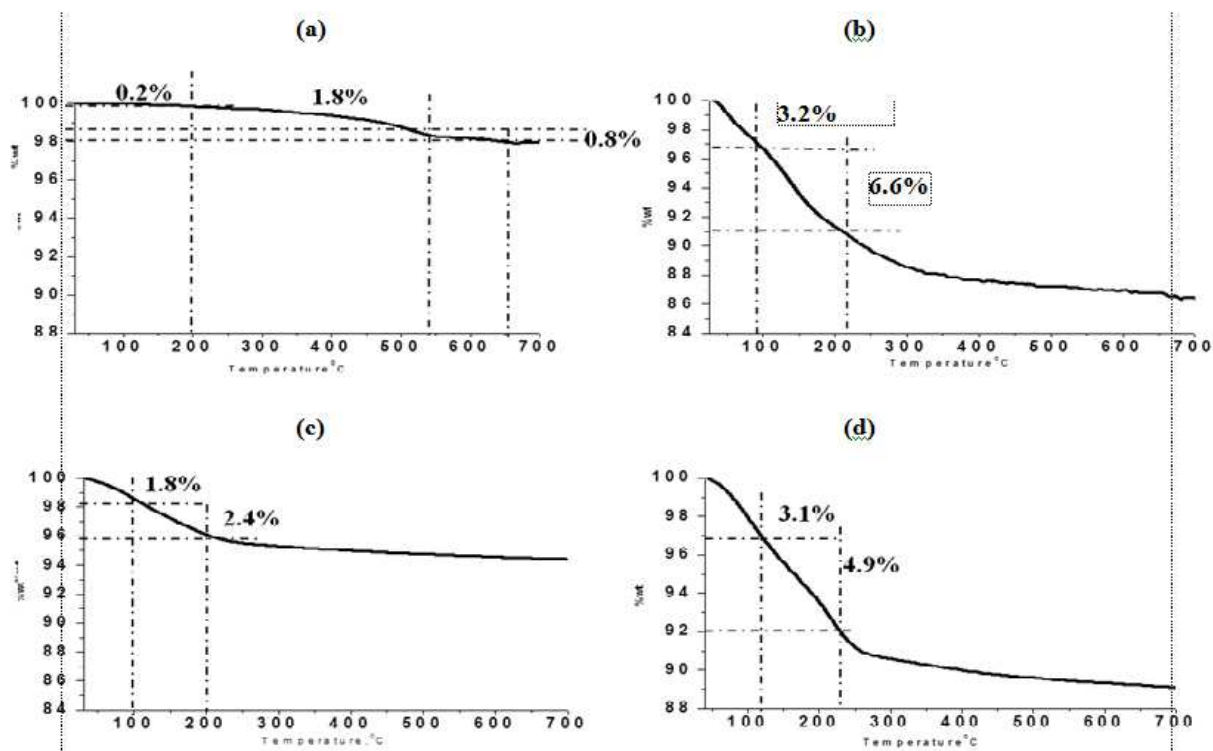


Figure 4. Thermograms of Fly Ash and the Product Obtained at Varying Temperature of Gel Formation

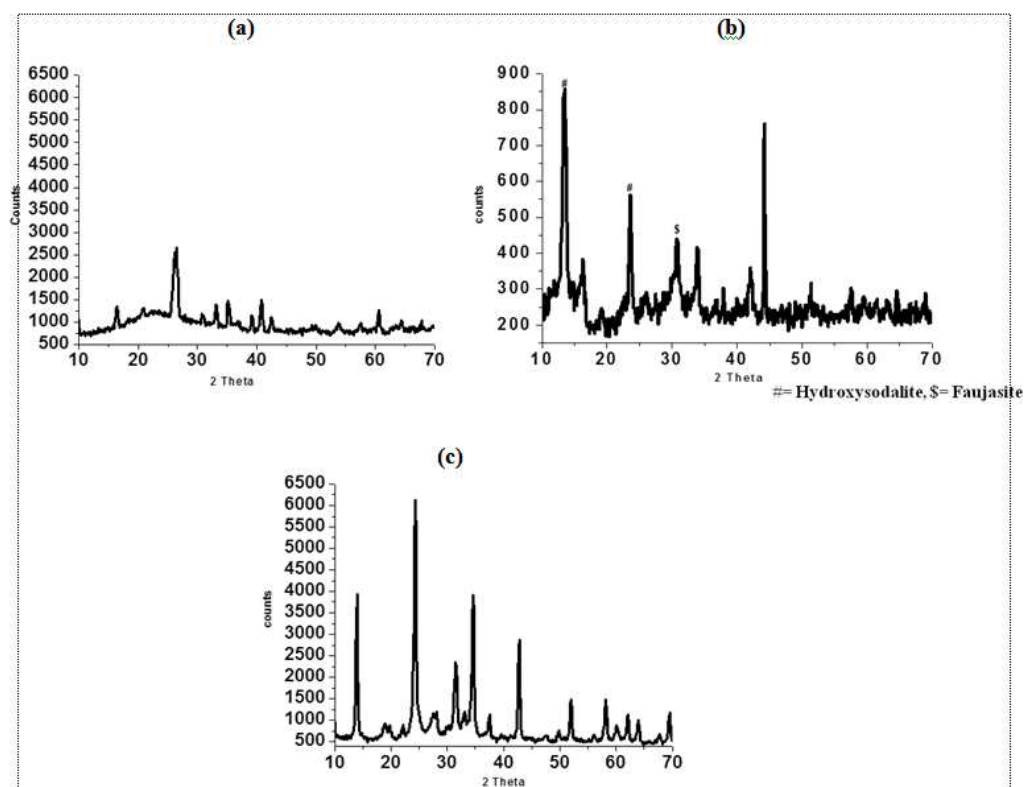


Figure 5: XRD Patterns of Fly Ash and the Product Obtained at Various Time Periods of Gel Formation

Influence of Gel Formation Temperature and Time on Complete Conversion of Fly Ash in to a Framework Aluminosilicate Utilizing Alkaline Hydrothermal Synthetic Methodology

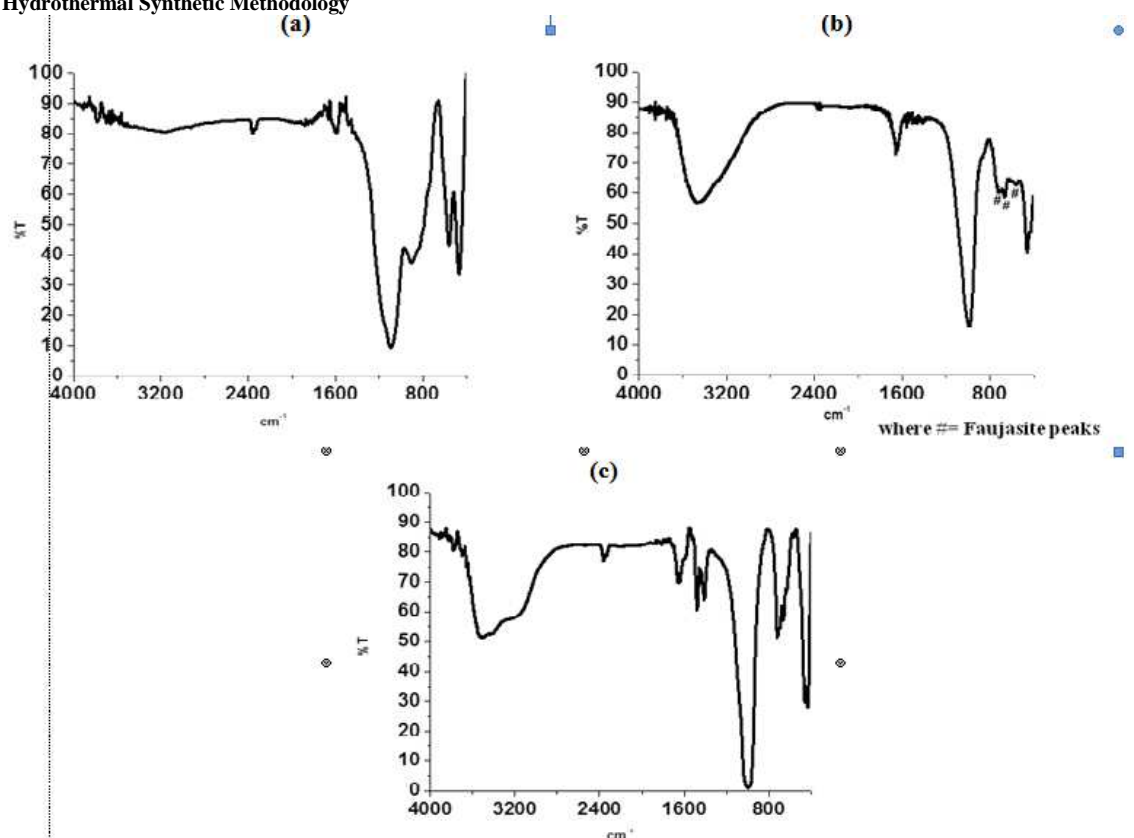


Figure 6: FT-IR Patterns of Fly Ash and the Product Obtained at Various Time Periods of Gel Formation

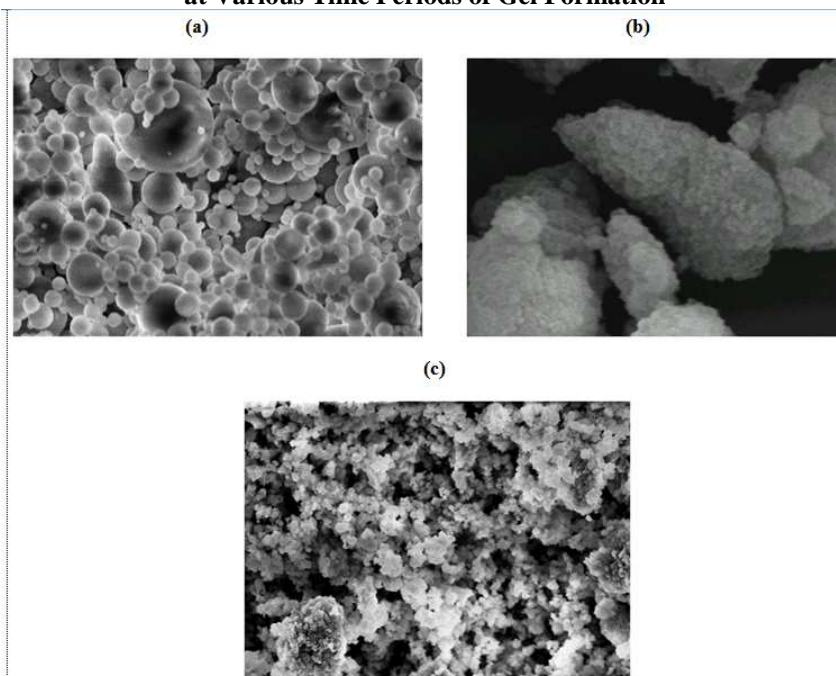


Figure 7: SEM Patterns of Fly Ash and the Product Obtained at Various Time Periods of Gel Formation

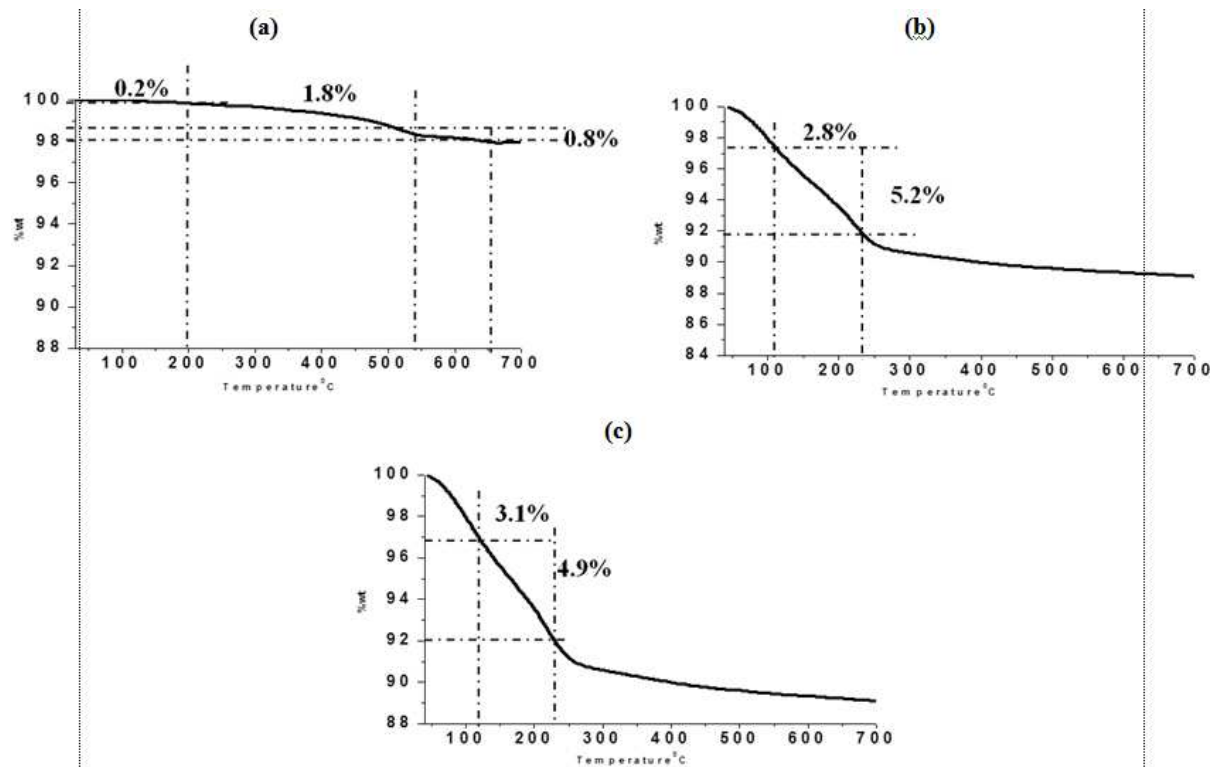


Figure 8: Thermogrms of Fly Ash and the Product Obtained at Various Time Periods of Gel Formation

Table 1: Selected Parameters for Synthesis, Variation in Gel Formation Temperature

| Product Code | [Alkali] | Initial Mixing | | Ageing | | Curing | |
|--------------|----------|----------------|------|--------|------|--------|------|
| | | Temp. | Time | Temp. | Time | Temp. | Time |
| S-02 | 3M | 30°C | 48 | 30°C | 48 | 40°C | 72 |
| S-03 | 3M | 60°C | 48 | 30°C | 48 | 40°C | 72 |
| S-04 | 3M | 80°C | 48 | 30°C | 48 | 40°C | 72 |

Table 2: Vibrational Frequencies of Fly Ash

| Vibrational Frequencies (Cm ⁻¹) | | Assignments |
|---|-----------------|---|
| Observed Values | Reported Values | |
| 1096(s), 906(sh) | 1135-1080 | Si/Al-O, Asym Stretching Quartz & Mullite |
| Broad Shoulder | 792-700 | Si/Al-O, Sym. Stretching Quartz & Mullite |
| 560 (s), 458(s) | 560-550, 460 | Si/Al-O, Bending Mullite, Quartz |

Table 3: Selected Parameters for Synthesis, Variation in Gel Formation Time

| Product Code | [Alkali] | Initial Mixing | | Ageing | | Curing | |
|--------------|----------|----------------|------|--------|------|--------|------|
| | | Temp. | Time | Temp. | Time | Temp. | Time |
| S-05 | 3M | 80°C | 24 | 30°C | 48 | 40°C | 72 |
| S-06 | 3M | 80°C | 48 | 30°C | 48 | 40°C | 72 |